

# Geochemical Speciation of Metal Ions in the Leachate of Tailings Treated with Synthetic Rain Water

Elvis Fosso-Kankeu\*, Brad Barlow, Nico Lemmer and Frans Waanders

**Abstract**—Prediction of the possible mobilization of metals from tailings and their speciation is necessary for implementation of site specific management strategies. In this study 11 different tailings samples were collected near the city of Krugersdorp at an abandoned mine tailings dam. Samples from the top, middle and bottom of the tailings were assessed for twelve consecutive weeks through means of column leaching experiments. The parameters analysed for assessment of speciation of metal pollutants were pH, electrical conductivity (EC), oxidation redox potential (ORP), alkalinity, acidity, major anions (sulphate, chloride). Mineralogical- and chemical characterization of the tailings sample were conducted using X-ray diffraction (XRD) and X-ray fluorescence (XRF) respectively. Metal analysis was performed by means of inductively coupled plasma optical emission spectroscopy (ICP-OES). Speciation of metal ions was conducted through the use of PHREEQC modelling software. The main objective of this study was to determine the impact of acid rain on the mobility of metals in the tailings. XRD results showed that the bottom and middle of the tailings are dominated by quartz whilst the top was found to be dominated by alunite. The XRF results showed that the major oxide minerals were  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ . The ICP analyses showed that Ca and Na were most likely to leach from the tailings; the major metal pollutants leached were Al, Ca, Fe, Mg, Mn and Na. Synthetic acid rain had more effects on the metal mobility and the oxidation of mineral sulphides compared to samples treated without the acid rain. pH levels varied slightly from treated- and control samples. The PHREEQC results showed that the heavy metals tend to form free ionic species with samples treated with synthetic acid rain, consequently increasing the bioavailability of the heavy metals and therefore the toxicity levels in water bodies exposed to tailings.

**Keywords**— Mine tailings, metal mobility, synthetic acid rain water, ORP, pH, synthe Sulphate, XRD, XRF

## I. INTRODUCTION

Abandoned mine tailings pose a threat to the environment when toxic heavy metals leach into the ground layer below as a result of acid mine drainage (AMD) [1 - 8]. AMD occurs primarily when pyrite is exposed to oxygen. This oxygenation process degrades the quality of exposed surface water and lower

its pH values [1]. Acid mine drainage in South Africa occurs primarily from active and abandoned mine tailings exposed to rain and other weather conditions. The process starts as oxygenated rain water falls on the

Exposed mine tailings, causing oxidation of pyrite ( $\text{FeS}_2$ ) to occur [10 - 12]. The pyrite undergoes oxidation in a two-stage process, the first producing sulphuric acid and ferrous sulphate and the second orange-red ferric hydroxide and more sulphuric acid. The sulphuric acid then percolates through the tailings and dissolves the heavy metals in transit making these heavy metals increasingly soluble. Effectively these heavy metals emerge from the base of the tailings dump to add to the local ground water as a pollution plume [13]. The degree of mobilization of metals from tailing dumps by acid rain is influenced by the nature or properties of the tailing dumps; implying that the deriving environmental impact will be site specific. It is therefore important to predict the possible mobilization of metals from a given tailing in order to implement proper management strategy for the mitigation of pollution. Mobilization and dispersion of heavy metals as a result of AMD influenced by normal rainfall (pH 5.6) have been studied. It is the effects of acidic rainfall (4.0 - 4.6pH [14]) on exposed mine tailings that is of environmental concern. Prior to oxidation sulphuric- and nitric acid is present within the water molecules of acidic rain. The addition of sulphuric acid prior to oxidation of pyrite increases the overall concentration of sulphate within the tailings dam which could consequently increase the solubility and with that the bioavailability of heavy metals within the tailings [15]. In addition the speciation of metals within the tailings may tend to differ from normal rainfall conditions, this will affect the dispersion as it is dependent on the speciation of metals. Consequently if metals tend to occur mainly in the free hydrated form as opposed to complex metal form the mobility of heavy metals will increase as well as toxicity. The occurrence of metals in surface and ground waters is of concern as these can cause carcinogenic effects and toxicity in aqueous system and to people using such waters [16 - 27]. The purpose of this study is to determine the impact of acidic rainfall on the *mobility* of heavy metals within the tailings as well as to effectively determine the potential of the *dispersion* of metals released from the tailings.

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## II. EXPERIMENTAL SETUP AND MODELLING

### A. Materials

Soil samples were collected at a depth between 0-30 cm, using an auger drill, from abandoned gold mine tailings near Krugersdorp in the province of Gauteng, South Africa. A total of eleven samples were collected at different elevations of the tailings dam to ensure variations in the sampled soil. The soil samples (from each layer) were then mixed together to form three complex samples representing the top, centre and bottom of the tailings. Samples were then dried using heating ovens at 110 °C for a period of 3 hours. Samples were then stored in tight seal plastic containers before use.

### B. Mine Tailings Characterization

Characterization of the mine tailings was conducted by determining the chemical- and mineralogical composition as well as the particle size distribution (PSD) of the representative samples for the top, centre and bottom of the tailings dam. Prior to analysis, 40 g of the samples representing each layer were weighed off and then crushed to a particle size of 75 µm. The mineralogical composition was then determined using a Philips X'Pert pro MPD X-ray diffractometer for the X-ray diffraction (XRD) analyses. The chemical composition was determined using a MagiX Pro & Super Q X-ray Fluorometer for X-ray fluorescence spectrometry (XRF). The particle size distribution for each representative layer was determined using Malvern Master-sizer equipment.

### C. Synthetic Acid Rain Production

Before soil samples could be packed in the leaching columns synthetic acid rain was produced using 98% sulphuric acid and 55% nitric acid. The solution used for the production of synthetic acid rain was in a 3:2 mL ratio of sulphuric acid to nitric acid. An aliquot of the acid solution was added to 100 mL distilled water which was used as a buffer solution to produce synthetic acid rain by adding 4 mL of the buffer solution to 500 mL of distilled water till a pH value of 4.2 was observed using a Hanna instruments 8424 pH meter. This experiment was repeated until 8 L of synthetic acid rain was produced.

### D. Column Leaching Experiments

The physicochemical parameters were analysed for a twelve week column leaching period. Twelve columns were used to conduct the experiment in triplicate for each representative layer. The remaining three columns were used for control samples. The columns were each packed with 800 g sample mixed with 300 mL synthetic rain water and distilled water for control samples beforehand. Each sample was then topped up to 10 cm above the packed material. The leachate of each column was then measured weekly for pH, temperature, electron conductivity (EC) and oxidation redox potential. A pH meter

with an analytical electrode (HI8424) was used for pH, temperature and ORP measurements. A conductivity meter with an electrode was used to measure the EC. Sulphate concentration was measured monthly with a COD and Multiparameter Photometer HI 83099. The leachate of each sample was thrown back at the top of the column after measurements were noted.

### E. Titration Tests

The acidity was measured through titration method with 0.02 N sodium hydroxide (NaOH) where 0.02 N sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) was used as standardization procedure. The alkalinity on the other hand was measured through titration method with 0.1 N sulphuric acid (H<sub>2</sub>SO<sub>4</sub>). For chloride ion concentration, titration was conducted using silver nitrate (AgNO<sub>3</sub>) and potassium dichromate (K<sub>2</sub>CrO<sub>4</sub>) as indicator.

### F. Metal Analyses

To analyse the metal concentration, 3 mL of each sample was first stored in the fridge at 4°C, then 1 mL of each sample was diluted by a dilution factor of 10 using distilled water and then stored in a lab fridge prior to analysis. Metal analysis was then conducted using inductively coupled plasma-optical emission spectrometry (ICP-OES).

### G. Speciation Modelling

To determine the aqueous speciation of the major metal ions in the leachate of tailings, AQUACHEM software was used interfaced with PHREEQC (program version 3.136-9191.) modelling software. For the purpose of this report the database Minteq.v4.dat was used. Before the input data could be used the ORP had to be calculated by adjusting with a correction factor obtained from Field Measurements of Oxidation-Reduction Potential [28], this value is then used to calculate the pe value using the following equation:

$$pe = E_h / (0.059) \quad (1)$$

pe denotes the negative logarithm of the electron activity.

E<sub>h</sub> denotes the oxidation redox potential of the sample.

The following input data was then used for speciation calculation: pH, Eh (pe), temperature and determined concentration of metals, Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup>.

## III. RESULTS AND DISCUSSION

### A. Particle Size Distribution

The particles size distribution for each representative layer is displayed in Fig. 1, Fig. 2 and Fig. 3. As is evident from the results the particle sizes from the bottom and the top of the tailings dumps range from 1 µm to 1000 µm. However the result obtained for the middle of the tailings indicate that the particle sizes range between 1 µm and 400 µm. Smaller particles have larger surface areas than larger particles, thus a larger area is left

exposed to the effects of AMD making it more likely for metals to be released in higher concentrations [29]. This indicates that metals are much more likely to leach from the middle representative sample and acidity likely to form than the other two representative layers.

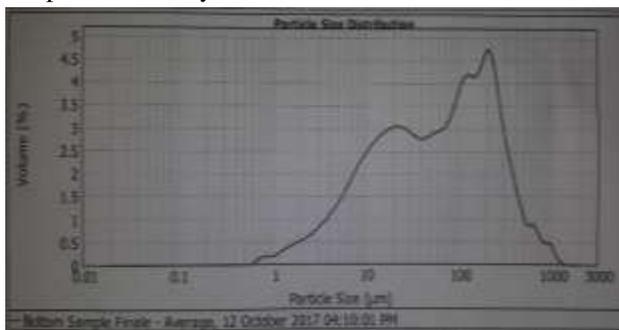


Fig. 1: Particle size distribution of the bottom tailings sample

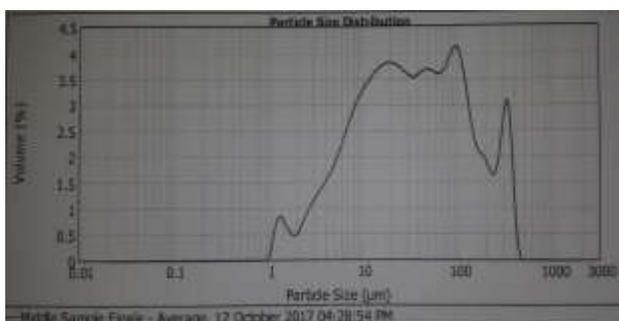


Fig. 2: Particle size distribution of middle tailings sample

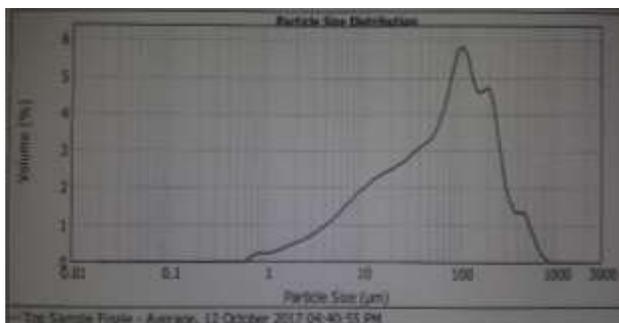


Fig. 3: Particle size distribution of top tailings sample

**B. Mineralogical and Chemical Composition**

TABLE 1:  
MINERALOGICAL COMPOSITION OF TAILINGS SAMPLES

Minerals	Weight % in sample layers		
	Bottom	Middle	Top
Quartz	77.60	84.87	-
Coesite	15.58	-	30.26
Iron Sulphide	0.17	4.92	-
Hematite	-	5.30	-
Aluminite	-	-	39.63
Aluminum Iron	-	-	27.68
Rozenite	-	-	1.56

XRD analyses were performed to evaluate the mineralogical composition of the representative tailings samples. The XRD results show that the bottom and middle of the tailings

comprised mainly of quartz. Other mineral such as amaranite, coesite, hematite and gypsum were also present. The top of the tailings was mainly comprised of aluminite coesite and aluminum iron.

An XRF analyses is preformed to evaluate the chemical composition of the representative tailings samples. The XRF results determined that the dominant oxide present in each tailings sample is SiO<sub>2</sub>. The other dominant oxides were identified as Al<sub>2</sub>O<sub>3</sub>, CaO, Fe<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O and SO<sub>3</sub>.

TABLE 2: CHEMICAL COMPOSITION OF TAILINGS SAMPLES

Samples	Mass & in sample layers					
	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	SO <sub>3</sub>	K <sub>2</sub> O	CaO	Fe <sub>2</sub> O <sub>3</sub>
Bottom	6.50	81.32	2.94	0.70	1.10	6.44
Middle	8.97	70.62	1.36	0.73	0.43	16.61
Top	9.08	84.50	1.50	0.45	0.71	2.74

**C. pH Results**

Fig. 4 shows the overall change in pH levels for each of the representative layers over the 12 week leaching period. The control samples, which were treated with distilled water, are displayed as BC, MC and TC for bottom-, middle- and top control samples respectively. The experiments were conducted in triplicate and the average values for the bottom (BS), middle (MSI) and top (TS) samples treated with synthetic acid rain were used for analysis as the standard deviation for these samples fell within the 10% margin.

Acidic condition was observed for the bottom and middle of the tailings with pH values of 2 and 3 whilst the top representative layer displayed alkaline conditions with pH values between 7 and 8.

The experimental results and control for each representative layer displayed the same behaviour as the trends are relatively the same. Although the bottom is dominant in sulphate concentrations as seen in **Error! Reference source not found.**, the oxidation rate tends to be very slow as the pH values of the bottoms samples (BC & BS) displays an almost constant trend as a slight decrease was observed over the 12 week period.

The pH values for the middle representative layer tends to vary as an increase in pH for both MC and MS from weeks 1 to 4 is observed. Buffering is dominant for the middle samples (MC and MS) until oxidation occurs as pH value decreases from weeks 5 to 12, first at a very slow rate and then sudden increase is observed between weeks 9 and 12.

The pH levels for the top of the tailings sample shows a constant increase for the 12 weeks leaching period, this can be due to the high concentration of Ca and Mg, observed through ICP analyses of metal ions, which contributes to an increase in pH [30]. This also corresponds with the increase in alkalinity measured for the top tailings sample over the 12 weeks period.

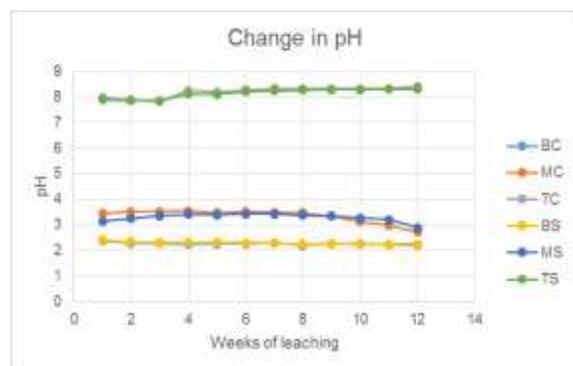


Fig. 4: Change in pH levels

#### D. Speciation Results

The five major heavy metal pollutants were analysed for the leachate of tailings treated with synthetic acid rain and for the control samples using PHREEQC workbench software. The workbench software effectively calculates and reports the speciation of the heavy metal pollutants in terms of their free hydrated ions and inorganic complexes [31]. These results are important as inorganic complexes tend to decrease the concentration of free aqua metal ionic species. Effectively this leads to a decrease in bioavailability and with it a decrease in toxicity levels. The PHREEQC results are displayed in Table 3. The dominant species of iron in the bottom and middle representative layers were in the free hydrated form of  $\text{Fe}^{+2}$  ranging from 49.96-78.93% for synthetic rain samples and 55.13-64.23% for control samples. The top tailings layer was also dominated by the free hydrated species  $\text{Fe}(\text{OH})_2^{+}$ . Complex hydroxide and carbonate species are also present ( $\text{Fe}(\text{OH})_4^-$  and  $\text{FeHCO}_3^+$ ) but at very low concentrations. Due to the high amount of free ions, iron is easily able to disperse in surface water causing an increase in toxicity. Magnesium was mainly comprised of the free ionic species  $\text{Mg}^{+2}$  and the complexed species  $\text{MgSO}_4$ . Other complex carbonate species of Mg are also present ( $\text{MgHCO}_3^+$  and  $\text{MgCO}_3$ ) but to a low extent. The speciation modelling predicted that manganese would mostly be soluble as a free ion species  $\text{Mn}^{+2}$  with a percentage range 58.29-82.56% for species treated with synthetic acid rain and 62.96-70.51% for the control samples. Manganese was also available in high concentrations of the complex species  $\text{MnSO}_4$ . Nickel was most abundant as a free hydrates species ( $\text{Ni}^{+2}$ ) for the acidic rain samples and as a complex ion ( $\text{NiSO}_4$ ) for the control samples. Other species of Ni predicted were  $\text{NiCl}^+$  and the carbonate species  $\text{NiCO}_3$ . Zinc follows the same trend as Ni with the acidic rain samples being more abundant as a free ionic species ( $\text{Zn}^{+2}$ ) and the control samples as a complex species ( $\text{Zn}(\text{SO}_4)_2^{-2}$ ). The free ionic species were the greatest for samples treated with synthetic acid rain. Thus for tailings exposed to acidic rainfall with the addition of acids prior to oxidation increases the bioavailability of metallic species within the tailings consequently increasing the toxicity levels in ground

water as these heavy metal species are mobilized from the tailings dumps.

TABLE 3: SPECIATION RESULTS FOR MAJOR HEAVY METAL POLLUTANTS IN THE LEACHATE OF TAILINGS

Element	Species	%Range for synthetic rain samples	%Range for control samples
<b>Fe(2)</b>	$\text{Fe}^{+2}$	49.96-78.93	55.13-64.23
	$\text{FeSO}_4$	21.31-48.89	38.34-44.88
	$\text{FeOH}^+$	0.21-1.06	0.39-0.82
	$\text{FeHCO}_3^+$	<1	<1
	$\text{Fe}(\text{OH})_2^+$	0-81.5	0-77.03
<b>Fe(3)</b>	$\text{Fe}(\text{OH})_3$	0-54.13	0-31.25
	$\text{Fe}(\text{OH})_4^-$	0-12.74	0-13.44
	$\text{FeOH}^{+2}$	<1	<1
	$\text{Mg}^{+2}$	30.9-78.67	36.06-61.38
<b>Mg</b>	$\text{MgSO}_4$	21.33-69.13	39.29-63.90
	$\text{MgHCO}_3^+$	<1	<1
	$\text{MgCO}_3$	<1	<1
	$\text{MgOH}^+$	<1	<1
<b>Mn(2)</b>	$\text{Mn}^{+2}$	58.29-82.56	62.96-70.51
	$\text{MnSO}_4$	15.89-41.33	28.32-37.29
	$\text{MnCl}^+$	0-1.5	0.11-1.17
	$\text{MnCl}_2$	<1	<1
	$\text{NiSO}_4$	22.04-69.52	40.89-64.98
<b>Ni</b>	$\text{Ni}^{+2}$	28.34-72.18	33.44-57.61
	$\text{NiCl}^+$	0.32-3.85	0.52-3.19
	$\text{NiCO}_3$	0-2.65	0.98-3.88
<b>Zn</b>	$\text{Zn}(\text{SO}_4)_2^{-2}$	2.59-69.57	11.36-74.46
	$\text{ZnSO}_4$	13.17-40.61	17.29-39.64
	$\text{Zn}^{+2}$	4.91-71.24	8.16-49.60

#### IV. CONCLUSION

The PSD results for the tailings samples shows that the particles tend to be present in larger sizes in the top and bottom of the tailings (1-1000  $\mu\text{m}$ ) whilst the middle representative layer contained smaller samples in the range 1-400  $\mu\text{m}$ . The data analyses of the physicochemical parameters shows that there is a difference between the measured values of the leachate of the tailings treated with synthetic acidic rain water and distilled water (control). The pH levels for the top and bottom of the samples displayed almost identical results with slight variations. The middle tailings samples had larger variation to the control samples. The pH is lower for the samples treated with synthetic acid rain. PHREEQC results showed that heavy metals in the tailings treated with synthetic acid rain were abundant in the free ionic form which enhances the mobility of these metals contributing to increased levels of pollution. Control samples were more mostly contained the complex form, however some free ionic species were also predicted.

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